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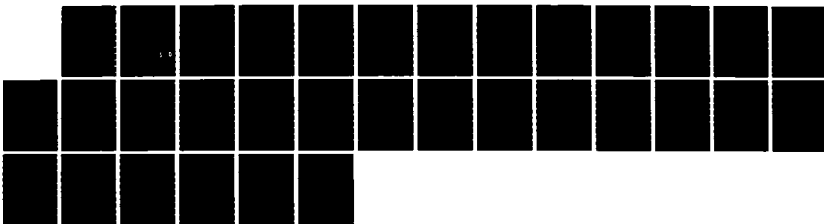
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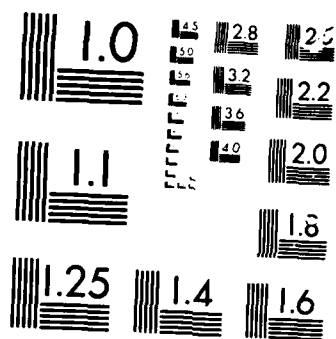
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Ionic Conductivity in Solid, Crosslinked Dimethylsiloxane-
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by

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below the "central" glass transition temperature T_g .

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Ionic Conductivity in Solid, Crosslinked Dimethylsiloxane-
Ethylene Oxide Copolymer Networks Containing Sodium

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Abstract

The preparation of an ion-conducting elastomeric solid based on a dimethylsiloxane-ethylene oxide copolymer complexed with a sodium salt is described. ^{23}Na Nuclear Magnetic Resonance measurements reveal the presence of both bound and mobile sodium species throughout the temperature range -120 to 100 C. Electrical conductivity measurements over a similar temperature range are found to be consistent with the configurational entropy model for transport, with a T_0 parameter about 50 C below the "central" glass transition temperature T_g .

Introduction

An intriguing and potentially technologically useful class of solid electrolytes consisting of alkali salts dissolved in polymer hosts has recently received a great deal of attention.⁽¹⁾ Much of the effort to date has focused on poly(ethylene oxide) (PEO) as the host.^(2,3) Although these materials are among the most highly conducting in their class (10^{-3} - 10^{-4} $\Omega\text{-cm}^{-1}$) at 100C), their attractiveness is limited by the high degree of crystallinity of the complex (the ion transport occurs most effectively in the amorphous phase), and the relatively low value of the electrical conductivity at room temperature. The latter property poses a serious threat to ambient temperature electrochemical device applications, most notably batteries.

The advent of polymer complexes with room temperature conductivities substantially higher than observed in Li-PEO complexes was highlighted by the synthesis and study of materials based on polyphosphazine, which appear to incorporate the "best of both worlds" with regard to flexibility of the phosphazine polymer backbone and the cation-solvating properties of ethylene oxide chains contained in the sidegroups. The chain flexibility at room temperature is related to the complex's low glass-transition temperature (T_g), some 30-50 C lower than in analagous PEO complexes.⁽⁴⁾ Another low T_g system that has received some consideration is based on co-polymers of dimethylsiloxane and ethylene oxide.^(5,6) In addition to enhanced electrical properties via higher room temperature conductivities, the low T_g materials generally possess favorable elastic characteristics

that can be exploited in electrochemical devices. Other motivations for exploring new compositions are associated with finding improvements in such characteristics as thermal and electrochemical stability and moisture resistance.

This paper reports the synthesis of a highly crosslinked poly(dimethylsiloxane-ethylene oxide) (PDMS-EO) copolymer network containing 11.5% by weight NaCF_3COO . The material has been studied by ^{23}Na nuclear magnetic resonance (NMR), electrical conductivity, and differential scanning calorimetry (DSC) measurements, the results of which are presented and discussed below.

Experimental Details

The sample preparation consisted of three steps: polymerization (chain extension of the prepolymer); complexing with a sodium salt; and crosslinking. Ethylene-dimethylsiloxane oxide ABA block copolymer (Petrarch; m.w. 1000-1500; ethylene oxide 75 to 80% by weight), and dimethylsiloxane-ethylene oxide block copolymer (Petrarch; m.w. 1120) were used as prepolymers. Triacetoxysilane and triethoxysilanes were employed as crosslinking agents, 3-isocyanatopropyltriethoxysilane (Petrarch) yielding the best results. Prepolymer(s) and crosslinker in 200 to 300% molar excess were dissolved in THF and polymerization was carried out at 70 C for about 5 hours while refluxing in open air. About 5% water was added as an initial polymerization catalyst for the silicone system. Subsequent addition of glacial acetic acid (1.0 molar with respect to the prepolymer) was found to improve

control over the polymerization reaction, although some compositions were prepared without it. A suitable sodium salt, purified by recrystallization from THF, 10 to 20% by weight of the composition (which is one salt molecule per 4 to 8 ethylene oxide units), was added at the end of the polymerization step. Another 300% molar excess of crosslinker was then introduced and the formulation was vigorously mixed at 70 C while allowing the solvent to evaporate at atmospheric or slightly sub-atmospheric pressure. It is important to evaporate most of the solvent before casting to avoid inhomogeneities and flaws in the solidified polymer composition.

The resulting highly viscous formulation was poured into a teflon mold to be cured by alkoxide condensation in two steps: precured for 15 to 18 hours at 60 C and atmospheric or slightly reduced pressure (about 600 mm Hg) and then cured for 4 to 6 hours at 110 to 115 C under reduced pressure (roughing vacuum) to remove the remaining solvent, catalyst and condensation by-products. The samples exhibiting the most favorable combination of homogeneity and elasticity were transparent, slightly colored (light brown) polymer films.

The samples on which the NMR, DSC, and conductivity measurements were performed utilized 200% molar excess of the crosslinking agent 3-isocyanatopropyltriethoxysilane during polymerization, with an additional 300% molar excess in the complexing and curing stage. Sodium trifluoroacetate (Aldrich) constituted 11.5% by weight of the complex, corresponding to 1 salt molecule per 8 ethylene oxide units. The precure was

performed at 60 C for 16 hours, and the final cure at 110 C for 6 hours. The sample was verified to be amorphous by X-ray diffraction and DSC. Excess water or solvent employed during polymerization and crosslinking did not appear to affect material properties, as substantiated by NMR measurements of the sample prior to, and following outgassing at 80 C for 48 hours in a roughing vacuum.

The NMR measurements were performed on a Novex pulsed NMR spectrometer interfaced to an IBM-PC computer, in conjunction with a Cryomagnet Systems superconducting magnet. The ^{23}Na spin-lattice relaxation (T_1) data were obtained at an operating frequency of 81 MHz, utilizing both inversion recovery and steady-state pulse sequences. The presence of two distinct lineshape components with vastly different T_1 's (details given later) allowed separate determination of their respective T_1 's by selective saturation and subtraction. The sample temperature (accurate to $\pm 2\text{K}$) was controlled by an N_2 flow system.

For conductivity determination, aluminum electrodes were vacuum evaporated onto the surfaces of the material in either a three-terminal or two-terminal configuration. The samples were about 1 mm thick and the electrodes about 4 mm in diameter. Measurements of the equivalent parallel capacitance, C , and conductance divided by the angular frequency, G/ω , were made at seventeen audio frequencies, 10x, 20x, 31.25x, 50x, and 100x Hz where $x=1, 10, 100$, and 1000 using a CGA-83 capacitance bridge, which is a fully automated, microprocessor controlled transformer

ratio arm bridge. The measurements were performed in vacuum using a Cryogenics Associates CT-14 dewar. The data were taken with the temperature held constant with approximately 60 minutes of equilibration time. The temperature was controlled using a Lakeshore Cryotronics DRC 82C controller and silicon diode temperature sensor. In general, the temperature stability was better than 0.005K as determined using a platinum resistance thermometer. The absolute temperature is probably accurate to on the order of 0.1K.

Differential scanning calorimetry (DSC) measurements were carried out using a DuPont 990 DSC. All the systems, bridge, temperature controller, and DSC were interfaced with Apple II microcomputers.

Results and Discussion NMR

The ^{23}Na absorption lineshape consists of a relatively narrow (0.5 - 5 kHz) line superimposed on a broader line (20 - 30 kHz) (both FWHM), throughout the temperature range -100 to +100C. The broad line is associated with the $+1/2$ to $-1/2$ central transition of spin- $3/2$ ^{23}Na (as indicated by the optimum pulse widths for the separate absorption components). The strength of the nuclear quadrupole interaction (of the order of 1 MHz) inferred from the second-order splitting implies that the corresponding Na nuclei reside at asymmetric sites for a time scale considerably greater than 10^{-6}s . The broad component is thus associated with a rigidly bonded configuration while the narrow component reflects a highly mobile and ionic disposition.

This assignment is strongly supported by the 2-3 order of magnitude difference in T_1 's shown in Fig. 1, a plot of T_1 vs. reciprocal temperature for both the broad and narrow lines. T_1 for the narrow line has a more pronounced temperature dependence than for the broad line, in addition to its substantially shorter timescale. Between the glass transition temperature (-50°C) and room temperature, the relaxation is approximately Arrhenius with an activation energy of roughly 0.1eV. A T_1 minimum appears just above room temperature.

It is important to note that motional processes governing spin-lattice relaxation do not necessarily play as important a role in ionic transport due to the vastly different length scales involved. Therefore it is not surprising that the conductivity temperature dependence, which obeys a VTF-type relation⁽⁷⁾ (details presented below), is qualitatively different from the Arrhenius T_1 behavior. The conductivity is, in general, strongly influenced by the concentration of mobile species, which is not obtainable from the data in Fig. 1. However, an important qualitative observation concerning the relative intensities of the broad and narrow lineshape constitutes as a function of temperature has been made. There is an increase in mobile Na intensity at the expense of "bound" Na intensity with increasing temperature, although both species are present at all temperatures between -120 and 100°C . This observation is reminiscent of similar phenomena reported in PEO-complexes⁽⁸⁾ and, in fact, most polymer systems that exhibit a coexistence of

amorphous and crystalline phases. The important distinction to be made here is that the title compound does not appear to have a crystalline phase, as verified by x-ray and DSC. Quantitative determination of relative concentrations via broad/narrow intensity measurements are complicated by the different $\pi/2$ -pulse widths for each component, although such measurements are currently in progress and will be presented in a later publication. With regard to material stability, exposure to ambient atmosphere for several weeks did not result in noticeable changes in either physical appearance of the sample, or in lineshapes and relaxation times.

The conductivity data were analyzed using standard complex impedance techniques allowing the determination of the bulk resistance as a function of temperature. A low temperature impedance plot is shown in figure 2. A single depressed arc is observed over the frequency range of measurement. The data were analyzed using a Cole-Cole distribution:⁹

$$Z^* = \frac{Z_0}{1 + (i\omega\tau_0)^{(1-\alpha)}} \quad (1)$$

The best-fit Cole-Cole result is shown in figure 2 along with the center of the circle, +, and the intercept which gives the bulk resistance, x. For the data shown in figure 2 the Cole-Cole parameter, α , is about 0.25. As temperature increases, less of a semicircle is observed with the onset of a typical slanted vertical line representing blocking electrode effects. In all cases, a best-fit of equation 1 to the data was obtained which

resulted in values for the bulk resistance of the materials.

These values were then used, in conjunction with room temperature geometrical measurements, to calculate the electrical conductivity from:

$$\sigma = Gt/S \quad (2)$$

where t is the thickness and S is the surface area. Thermal expansion is not included in the data analysis. The results of a typical data run are shown in figure 3. The curvature often observed for amorphous polymer systems is apparent. That the samples were amorphous is confirmed by the DSC results shown in figure 4. Consequently, the conductivity data were first analyzed via the VTF equation:⁷

$$\sigma = AT^{-1/2} \exp \left\{ -[E_a/k(T-T_0)] \right\} \quad (3)$$

with the adjustable parameters, A , E_a , and T_0 . A non-linear least squares fit of equation 2 to the data was carried out and Table I contains the best-fit parameters. Table II contains the results of the DSC studies.

The most interesting result is that T_0 is about -100 C which is about 50 C lower than the "central" T_g which was determined by DSC to be about -50 C. A similar result has also been recently reported for ion containing PPO.¹⁰ In that paper, it was stated that T_g was 30-40 C above T_0 . However, in that paper T_g was defined as the "onset" T_g . A similar result is obtained in the

present work since as is apparent from Table 2, the "onset" T_g is about 35 C above T_0 . Such results are not unexpected since $T_g - T_0$ is often on the order of 50 C for polymer systems.¹¹⁻¹³ Further, this phenomenon is consistent with the configurational entropy model^{14,15} where T_0 is interpreted as the temperature of zero configurational entropy which would be expected to occur at a much lower temperature than DSC T_g 's. However, this result disagrees with that of Bouridah et al.⁶ who find $T_0 \approx T_g \approx -60$ C. This is accompanied by a disagreement in the values of E_a in that Bouridah et al. report 0.069 eV while the corresponding value for the present work is about 0.1eV. A similar discrepancy, higher E_a and lower T_0 , has been noted previously in comparing reported VTF parameters for ion containing PPO.^{3,10} It was pointed out in reference 10 that because of the positions of E_a and T_0 in the VTF equation opposite variation will produce relatively little change in the conductivity. Thus, the discrepancies may be partially attributable to the data fitting techniques. Because of this possibility, further details concerning the present data analysis techniques are given.

In the present work and that of reference 10 the sum of the squares of the differences:

$$S = \sum_1 (\log_{10} \sigma_{\text{exp}_1} - \log_{10} \sigma_{\text{th}_1})^2 \quad (4)$$

was formed and explicit expressions for the three equations $dS/da_i = 0$ were derived and then solved numerically for three fitting parameters a_i . As a check of the procedure, the value of T_0 was fixed at -65 C and the remaining two parameters were best

fit. The value of E_a decreased from 0.1 to 0.052 eV and $\log_{10} A$ decreased from -0.5 to -1.73. However, the RMS deviation in $\log_{10} \sigma$ increased by over an order of magnitude from 0.0096 to 0.14. Consequently, it is concluded that the present data cannot be best fit by the VTF equation if T_0 is on the order of T_g .

In addition, since data are often presented in linear form, the results of the present work for sample #1 are replotted in figure 5 using a linear plot using the value of A from Table 1. Also shown is the best-fit straight line. The intercept at T_0 176K is obvious from the plot.

Next, the data were analyzed in terms of the WLF equation¹⁶:

$$\log_{10} \frac{\sigma(T)}{\sigma(T_g)} = \frac{C_1(T-T_g)}{C_2+(T-T_g)} \quad (5)$$

The resultant parameters are listed in Table II. The values of C_1 and/or C_2 are somewhat lower than the "universal" values of 17.4 and 51.6.

Finally, for completeness, the data were analyzed via the VTF eq. in the form:

$$\sigma = A' \exp -[E'_a/k(T-T'_0)] \quad (6)$$

The results are also listed in Table I. It is interesting that on the basis of the RMS deviation it is equation 3 which best fits the data.

SUMMARY

In summary, an ion conducting, highly crosslinked P(DMS/EO) copolymer network complexed with sodium trifluoroacetate has been synthesized. ^{23}Na NMR measurements demonstrate the simultaneous presence of a long T_1 bound sodium and a short T_1 mobile sodium. It is believed that the process of bound to mobile conversion of species with increasing temperature, observed qualitatively, is crucial to fast ion transport. Electrical conductivity has been measured and analyzed in terms of VTF and WLF equations. The most important result is that for the VTF equation T_0 is found to be about 50 C below the "central" T_g . This is consistent with the usual behavior of these quantities and is predicted by the configurational entropy model. As regards the WLF equation, the values of C_1 and/or C_2 are found to be slightly lower than the "universal" values.

Acknowledgements

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TABLE I. Best fit VTF parameters.

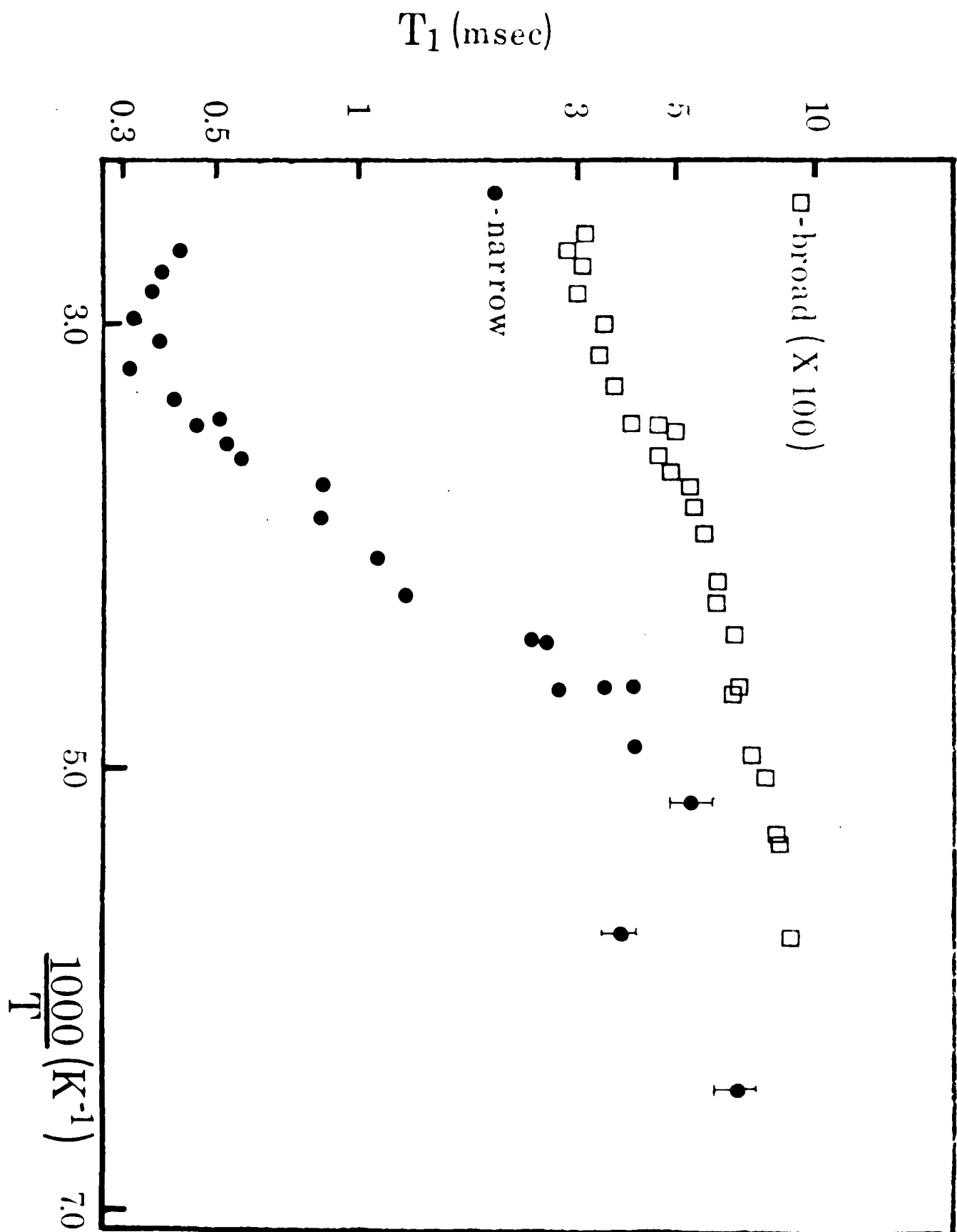
(Eq. 3)	RMS Deviation	$\log_{10}A$	E_a (eV)	T_0 (K)
Sample #1	0.0096	-0.50	0.103	176.4
Sample #2	0.0106	-0.61	0.104	171.8
(Eq. 6)	RMS Deviation	$\log_{10}A'$	E'_a (eV)	T'_0 (K)
Sample #1	0.0105	-1.90	0.098	178.2
Sample #2	0.0119	-2.00	0.099	173.6

TABLE II. Best fit WLF parameters.

	$T_g(K)$	$\log_{10} (T_g)$	C_1	$C_2(K)$	RMS Deviation
Sample #1 Onset	208	-18.4	16.5	29.8	0.0105
Central	223	-12.9	11.0	44.8	0.0105
End	238	-10.1	8.2	59.8	0.0105
Sample #2 Onset	208	-16.5	14.5	34.4	0.0119
Central	223	-12.1	10.1	49.4	0.0119
End	238	-9.7	7.7	64.4	0.0119

Figure Captions

- Figure 1. ^{23}Na T_1 vs reciprocal temperature for broad and narrow lineshape components.
- Figure 2. Typical low temperature complex impedance plot. The horizontal intercept yields the bulk resistance.
- Figure 3. Electrical conductivity vs reciprocal temperature, showing curvature characteristic of amorphous polymer systems.
- Figure 4. DSC plot, from which a "central" T_g of -50°C is deduced.
- Figure 5. Linear fit to the conductivity data, utilizing the value A from Table 1.



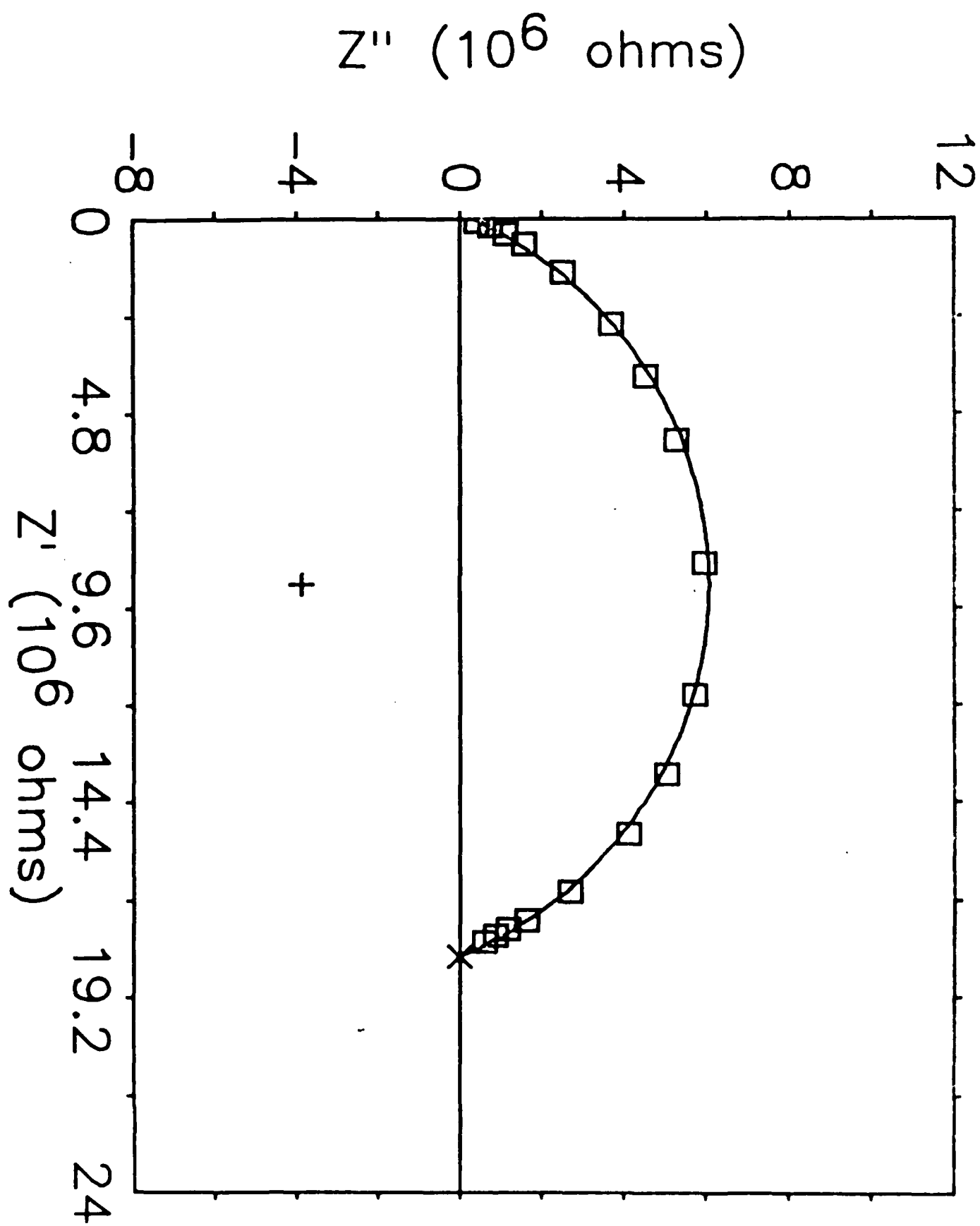
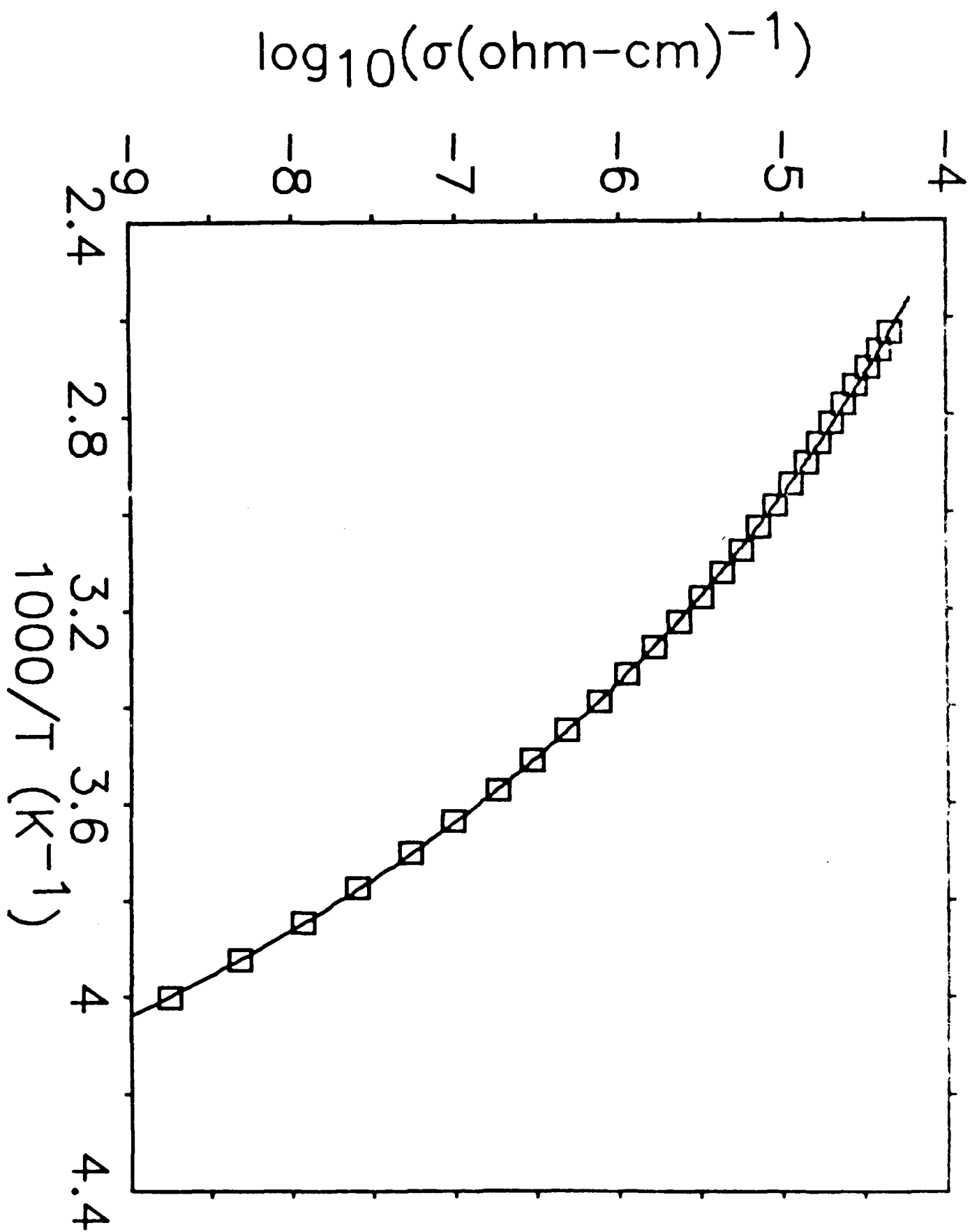
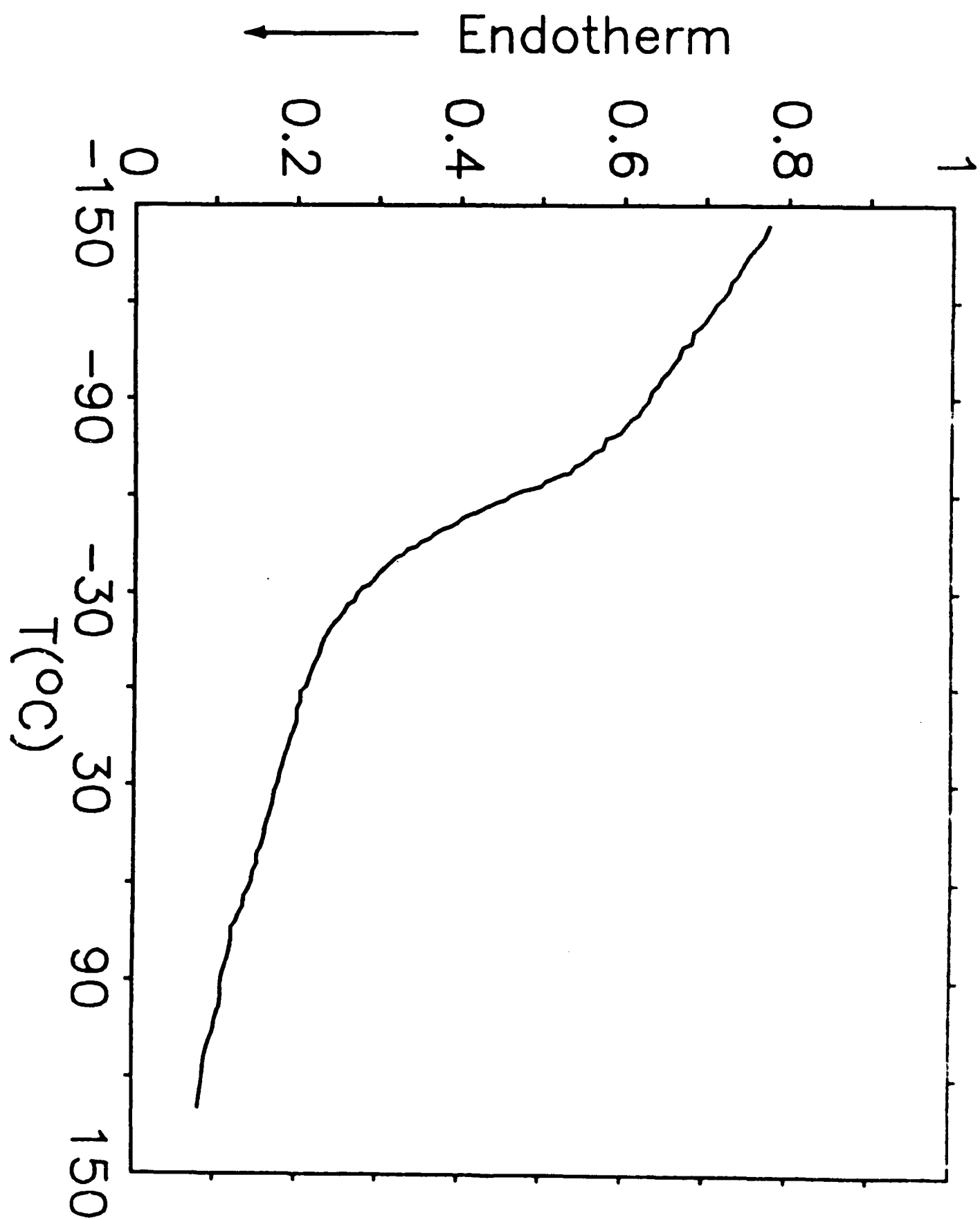
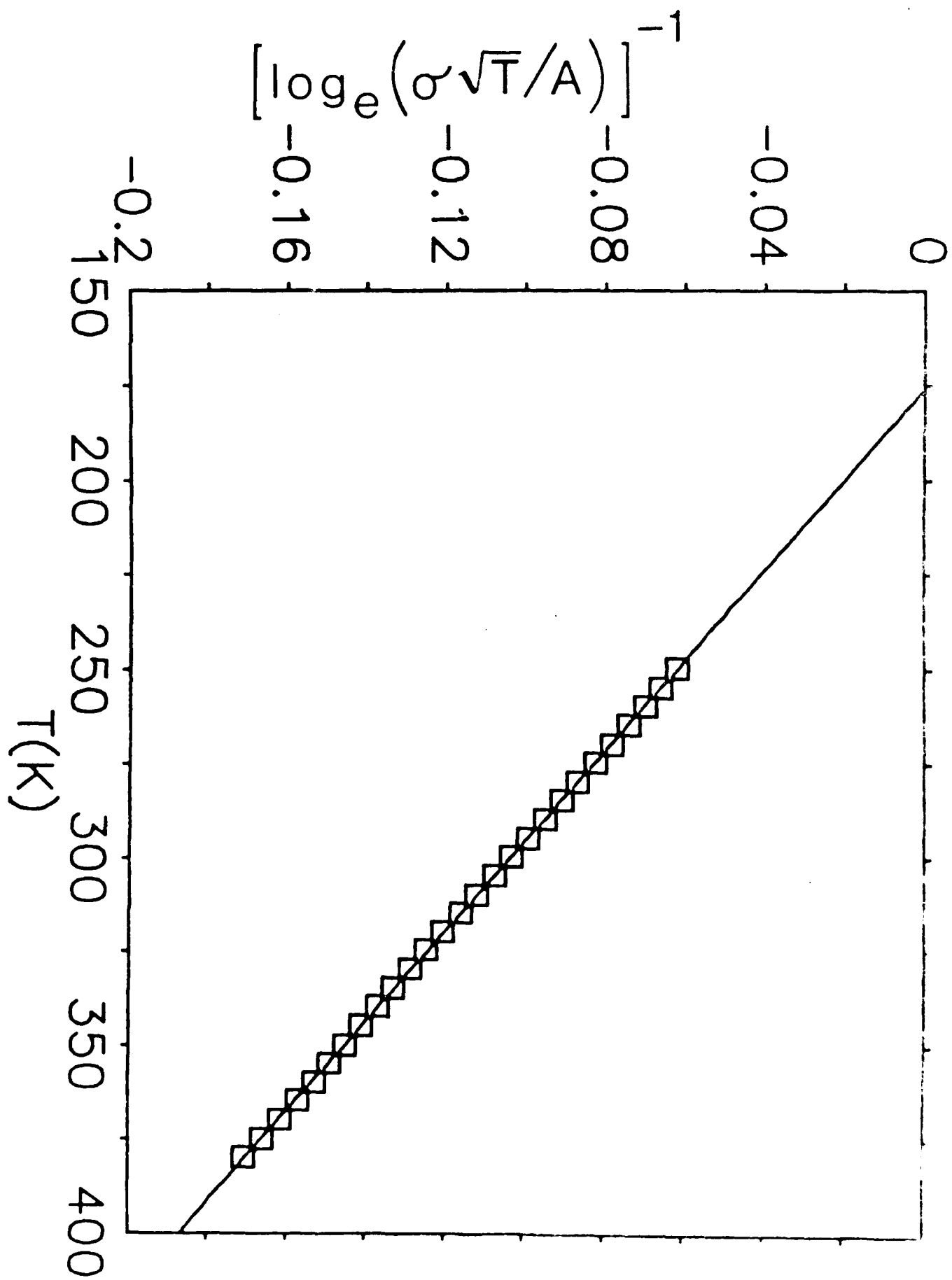


Fig. 2







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